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MANUFACTURE OF SUPERFINE ORGANIC FIBERS

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Chemistry Division

May 25, 1954



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ABSTRACT

Through a new process recently developed at NRL, it is now feasible to produce submicron organic fibers from a variety of thermoplastic materials. By this method an adjustable extruder forces a hot thermoplastic melt through a row of fine orifices into high-velocity dual streams of heated gas, usually air. The nozzle design provides for immediate resumption of attenuation following breaks, which are inevitable at submicron dimensions.

Fiber diameters are determined by four basic process variables: air and nozzle temperatures, air pressure, and polymer feed rate (or ram pressure), all of which may be controlled independently. Nylon, linear polyester, polytrifluorochloroethylene, silicone, polystyrene, and other fibers can be produced, but those polymers which possess low melt viscosities attenuate most readily. Materials such as polyvinyl chloride and polyacrylonitrile which do not melt or soften sufficiently below their decomposition temperature cannot be employed in this process. Proper balancing of all the variables yields fine fibers of good uniformity and quality.

By eliminating intermediate operations of fiber chopping and wet techniques of paper making, preparation of fabrics from thermoplastics is promising economically as well as technologically.

PROBLEM STATUS

This is an interim report; work on the problem is continuing.

AUTHORIZATION

NRL Problem C06-11
Project Nos. NR 406-110 and NE 120-704-1
Bureau No. S-1636

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MANUFACTURE OF SUPERFINE ORGANIC FIBERS

INTRODUCTION

A rather broad project was initiated at the Naval Research Laboratory in 1951 on the methods of production and the properties of organic fibers that were less than one micron in diameter. The so-called "fiber-forming" thermoplastic materials, which include nylon, linear polyesters, and polyethylene, were of particular interest in this investigation. This group is defined as polymers that undergo molecular orientation when they are cold-stretched. The only synthetic fibers available at that time were the ordinary textile-grade fibers, the finest of which were about 12 microns in diameter. At the outset, the object was simply to design an apparatus that would produce submicron fibers. The size was of primary importance because of its known effect on the efficient performance of filter papers. The form of the fibers, whether staple or continuous, did not at first matter, but early findings showed that fineness would play an important role in obtaining good fiber dispersion and strength for the final product. It was thought that a homogeneous material composed entirely of submicron synthetic fibers would have great usefulness for aerosol filtration as well as for dielectric insulation.

PRODUCTION METHOD

The apparatus developed for the attenuation of fine fibers is essentially an extruder that forces a hot melt of thermoplastic through a row of fine orifices and directly into two converging high-velocity streams of heated gas (usually air). The diagrams of Figure 1 illustrate the juxtaposition of the air streams and nozzles. The temperatures of the air and molten plastic are controlled separately, and the velocities of the air and molten plastic are also separately controlled. As shown in the cutaway view of the nozzle tip (Figure 1b), the individual orifices are actually slots which are milled into a flat surface and then matched with identical slots milled into a mating surface. When the two halves are placed together, they form a row of openings which, according to the preferred design, are 0.014 inch wide and 0.020 inch apart, center to center. As a result of this spacing, the flat surfaces between the grooves are 0.006 inch wide and form "dead" spaces that separate the fibers in the blast. The straight line arrangement of apertures permits any number* of orifices to be used in a nozzle. Generally, the orifices are set in groups of 50 in an inch with another inch separating the groups in order to facilitate proper clamping of the two nozzle halves.

The actual point at which a fiber is formed lies within the gas stream where the stream attains its peak velocity. Since the molten material issues from the nozzle directly into the confluence of the two air streams, the greatest amount of attenuation occurs at the point of exit. Thus, the orifice size has little importance provided it is

*This depends on the width of the desired fiber web

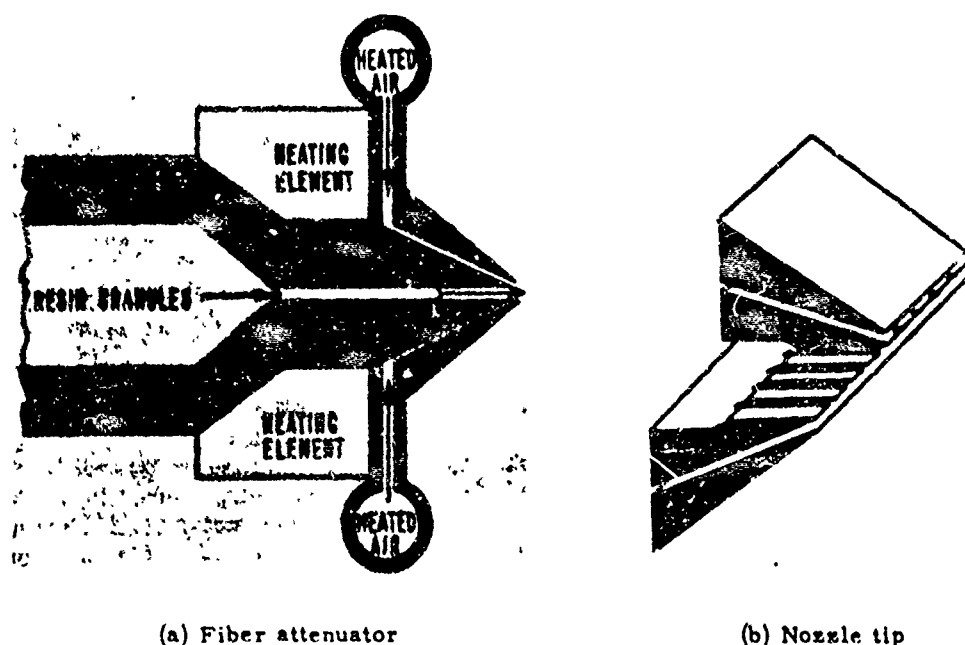


Figure 1 - Diagram of apparatus used to attenuate fibers

large enough to pass the melt without plugging. The orifice diameter and the orifice length determine the hydraulic pressure required to yield a given polymer feed rate. This pressure must be high enough to give good distribution and controllable flow to the plastic melt. Generally, an orifice length of one inch provides adequate back pressure for the typical hot melt. The velocities of the streams and their temperatures, the melt viscosity, and the size of the angle included by the two gas streams all combine to determine the extent of the zone of attenuation and the resulting fiber diameter. As a consequence of the cooling undergone by the air when expanding, the fibers are solidified upon reaching a point one to two inches away from the nozzle.

The dispersion of fibers produced by this process is illustrated with a photograph (Figure 2) taken by an illuminating flash of approximately one-microsecond duration. Here there were 15 streams of material being formed, and the gas velocity was held especially low so that the fibers would be large enough to be seen. Under normal conditions when submicron fibers are formed, their fineness and their greater movement resulting from higher gas velocities prevent the eye from seeing anything in front of the nozzle.

The collection method employed for the fibers is simply a process of pulling the air through a moving wire screen and thereby depositing the plastic on the screen as a random mesh of fibers. The type of network produced by this process is shown as an electron micrograph of a nylon fiber layer (Figure 3) resulting from a 0.1-second exposure to the air stream.

The nozzle angle, i. e., the angle included by the two air streams, is an important factor in nozzle performance. When this angle approaches 90° , a high degree of fiber dispersion results and the most random orientation is developed in the product. A nozzle angle of 30° , however, yields a greater number of parallel fibers deposited as loosely coiled bundles, but this angle also produces greater attenuation from a given gas velocity and, as a further advantage, has less tendency to form discontinuities in filament length. A prime feature of the entire design is the ability of the nozzle to continue the attenuation in

spite of breaks in the fiber, which are inevitable at submicron dimensions. When a break in a fiber occurs, the gas stream automatically resumes the attenuation, and only a small globule of nonfibrous material is formed.



Figure 2 - High-speed photograph showing production of nylon fibers



Figure 3 - Electron micrograph of nylon fiber layer

ATTENUATING CHARACTERISTICS OF VARIOUS THERMOPLASTICS

Among the factors that establish the ability of a given polymer to attenuate to a fine fiber are its melting point, viscosity-temperature characteristics, and surface tension which may be considered as incorporating intermolecular forces. The optimum operating conditions for a range of thermoplastics as given in Table 1 are simply those which yield the most continuous, shot-free fibers. Fibers with other diameters, however, can be made by merely changing any of the four basic variables, i. e., air temperature, nozzle temperature, air velocity (pressure), or polymer feed rate (ram pressure). Both polystyrene and polyethylene must be attenuated at temperatures well above their melting points.

The difference between air temperature and the melting temperature of polyethylene must amount to almost 800° F in order to reduce the melt viscosity sufficiently. Viscosities in the order of 1000 poise or less are desirable in fiber production by this process. When polyethylene and polytrifluorochloroethylene were attenuated, it was necessary to use special commercial grades that had low molecular weights. Any polyethylene that had a molecular weight greater than 12,000 remained too viscous for smooth attenuation even at high melting temperatures. Polyvinyl chloride and polyacrylonitrile were tried but both began to decompose at temperatures well below those necessary to achieve adequate fluidity.

TABLE 1
Attenuating Characteristics of Various Thermoplastics

Polymer	Melting Point (°F)	Nozzle Temp (°F)	Air Temp (°F)	Air Pressure (psi)	Ram Pressure (psi)	Fiber Diam (micron)	Rate* (lb/hr)
Nylon 6-6	510	680	700	50	100	0.3	0.11
Nylon 6-10	420	600	650	50	100	0.3	0.11
Nylon 6	410	600	650	40	80	0.7	0.24
Polyester (Dacron)	400	600	750	40	70	0.3	0.19
Polytrifluorochloroethylene	500	720	900	15	900	0.4	0.16
Polystyrene	300	700	840	20	220	1.0	0.17
Silicone	280	400	500	15	100	1.8	0.35
Polyethylene	220	700	600	10	300	2.0	0.24

* Production rate on a machine having 192 orifices.

Although operating temperatures as high as 800° F would normally be considered destructive to organic molecules, the exposure time at these temperatures is so short that negligible polymer degradation occurs. A short "holdup" time in the plastic melting stage also reduces degradation. The time is held small by a cold water jacket around the barrel of the ram-feeding mechanism. Being cooled in this way, the plastic does not melt until it moves up and contacts the heated face which leads it directly into the slot feeding the nozzle orifices.

The fiber diameters listed in Table 1 are estimates made on an optical microscope with dark field illumination. Correlations, however, were also made by the use of electron micrographs of the fibers and by an airflow resistance test. At a standard air flow of 85 liters per minute through a 100 cm² area of fiber mat, the air-resistance test yields a value, measured in mm of water, for the pressure drop. This resistance divided by the weight, in grams, of 100 cm² of the fiber is the value reported in Figure 4 as an indication of fiber diameter. The air resistance is related to fiber diameter by the following expression:

$$\text{Resistance} = \frac{C}{(\text{Fiber Diameter})^2}$$

where C is a constant whose value depends in part upon the porosity or density of the fiber mat. Since the porosity of the fiber product is not a constant value in this work, fiber size is indicated usually by the air resistance expressed in mm of water per gram of fiber.

To show the effects of the process variables on fiber size, a series of tests was made on polystyrene; they were based upon the optimum conditions given in Table 1 for this polymer (Figures 4 and 5). The results indicated that air resistance, or fiber diameter, was affected most sharply by nozzle temperature. When the nozzle temperature was below 680°F, the air resistance dropped rapidly thus indicating an increase in the average fiber diameter. This behavior is substantiated by observations that below a certain melt temperature a given thermoplastic will form large quantities of nonfibrous "shot." In fact, the normal method of establishing optimum operating conditions (Table 1) was to first find this critical nozzle temperature and then find the air velocity (pressure) that gave the minimum amount of shot. The air temperature and polymer feed rate were then adjusted to give the most freedom from fiber breaks. During the attenuation of polystyrene, the nozzle temperature must be carefully regulated because at a temperature of 720°F the fibers become almost too short to collect on the wire screen. The higher the nozzle temperature, the more difficult becomes the collection problem.

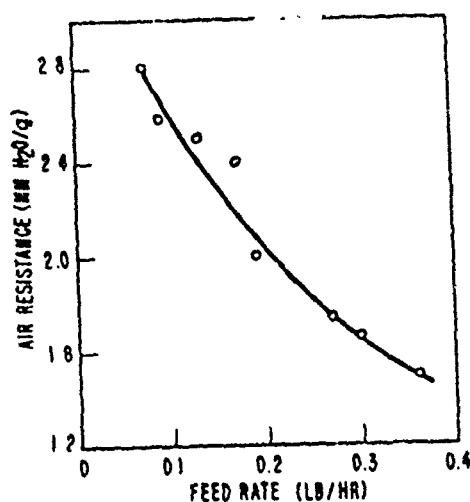


Figure 4 - Curve showing effect of polystyrene feed rate on fiber size

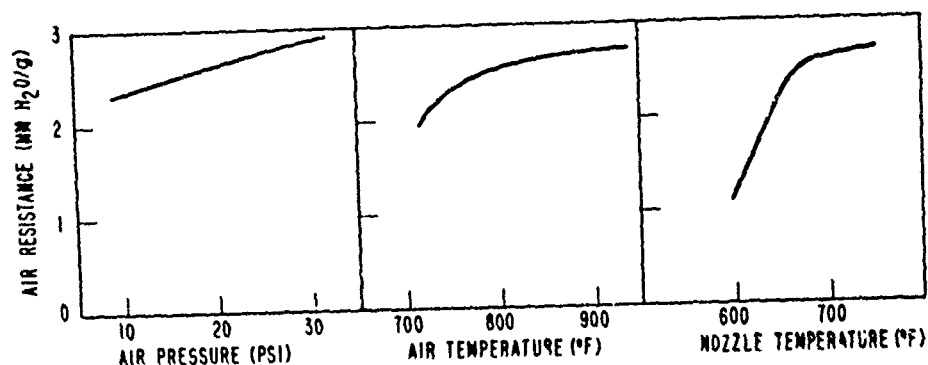


Figure 5 - Curves showing effect of process variables upon polystyrene - fiber size. A standard feed rate of 0.17 lb/hr was employed for each test and, except as indicated, all variables were maintained constant at the standard values: air pressure 20 psi, air temperature 840°F, and nozzle temperature 690°F.

The finest fibers with the most uniform diameters have been made from type 66 nylon. Figure 6 shows that the spread of fiber diameters may be contained within narrow limits provided temperatures and velocities are carefully balanced. This particular sample contains fibers whose diameters range between 0.11 and 0.22 micron. The achievement of



Figure 6 - Electron micrograph showing uniformity of nylon fiber diameter

this degree of uniformity, however, required an abnormally high air velocity - one which corresponded to a back pressure of 60 psi at the nozzle. Under such conditions, the production rate was slow and the collection of fibers rather difficult. High air pressures generally yield uniform and shot-free fibers but, at the same time, they make difficult the separation of fiber and air because of the excess volume of the latter.

PROPERTIES OF SUPERFINE FIBERS

Tensile Strength

The most outstanding characteristic of fine fibers is that when they are used in unbonded webs or mats they acquire tensile strength rapidly as the fiber diameter is decreased. This property is illustrated in Figure 7 where also the great influence of sheet density is strikingly shown. All of the tested sheets contained no binder, and the higher densities were obtained merely by a room-temperature pressing. During this work, an investigation of fiber diameters in the submicron range has been stressed because it represents a direct approach to the problem of obtaining homogeneity and freedom from pinholes in fibrous sheets. It is well-known that at least part of the strength and flexibility of cellulosic papers arises from the ability of regular cellulose fibers to subdivide or fibrillate and give a structure of submicron ultimate fibers. This idea is further supported by the fact that the flexibility of a fiber, expressed as the deflection under unit load, varies inversely with the fourth power of the fiber diameter. Another factor that favors decreased fiber diameter is the second-power relationship between the diameter of fibers and the number of them present in a unit of fabric. The number of contact points between fibers multiplies proportionally as the number of fibers in a unit volume increases, and as a result, the cohesive forces - the only forces holding these sheets together - are increased.

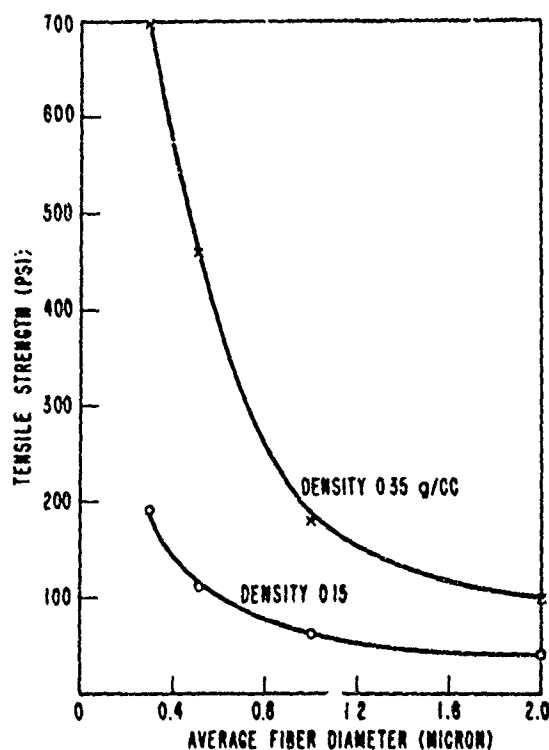


Figure 7 - Curves showing relationship between strength of unbonded nylon fiber mat and average fiber diameter

Molecular Orientation

The superfine fibers produced by this process might be expected to possess some degree of molecular orientation even though there is no cold-stretching performed on the fiber. When the fiber size is reduced from that of the orifice opening (0.010 inch) to a diameter of 0.3 micron, the fiber undergoes an 800-fold decrease in diameter, which corresponds to a 64×10^6 percent increase in length. It is reasonable then to expect molecular orientation. Furthermore, a fiber diameter of 0.3 micron approaches in size the average length of an uncoiled molecular chain. Nylon, for example, has an average length of about 1000 Å or 0.1 micron.

Efforts to show molecular orientation by x-ray diffraction have failed because a sufficient bulk of the fine fibers has never been aligned in a single direction. Attempts to work with single fibers have also failed. There is, however, an indirect indication of orientation within the fibers. A nylon (type 6-10) fiber web, 0.007 inch in thickness, was completely impregnated with a flexible polyester resin. When cured and tested without any fiber reinforcement, this resin possessed a 1700-psi tensile strength and an elongation of 300 percent. The same polyester when reinforced by the nylon mat possessed a tensile strength of 1850 psi but an elongation of only 50 percent. Since the broken edge of the test specimen showed no fibers protruding, it was assumed that the full nylon strength was realized. The stress that was applied to the plain polyester film at an elongation of 50 percent was 350 psi, and when this correction was subtracted from the strength of the reinforced specimen, a tensile value of 1500 psi resulted for the nylon mat alone. Two correction factors were then required to give an equivalent tensile strength for the individual fibers. The strength was divided by the factor $2/\pi$, which represents the average component of strength in the direction of test for fibers that are randomly distributed.

A further correction for the sheet porosity was made by dividing the strength by the ratio of the sheet density (0.17 g/cc) to the density of solid nylon (1.04 g/cc). The final approximation for the true fiber strength was then 14,000 psi. Since the strength of unoriented molded nylon (type 6-10) is 7000 psi and the elongation 135 percent, the increase in strength and decrease in elongation shown by these fibers are both changes that normally accompany the molecular orientation of a synthetic fiber. Because these conclusions are based upon a series of assumptions and approximations, they are necessarily only tentative.

PROCESS ECONOMICS

The principal costs involved in producing superfine fibers by this process are those incurred in heating and compressing large volumes of air. A typical requirement to produce one pound of 1.0-micron fiber might be 600 cubic feet of air or 50 pounds of air per pound of fiber. This air requirement seems to be proportional to the total length of fiber produced by a given weight of material. As a result, the requisite for a fiber having an average diameter of 0.3 micron is approximately 10 times greater, or 500 pounds of air per pound of fiber. If power at 1 cent per kilowatt hour is used, then the cost of heating air to 650°F would be approximately 0.043 cent per pound of air, and a liberal estimate for the cost of compressing would be 0.03 cent per pound of air. Based upon the production rate of the 0.3-micron fiber, costs would then be 17 cents per pound of fiber for heating and 12 cents for compressing. The total, 29 cents per pound of fiber, is small compared to the cost of the raw material, for example nylon. Of course, if only 1.0-micron fiber is desired, the costs would be about one-tenth the cost of 0.3-micron fiber, i.e., 3 cents per pound of fiber. Nylon type 3 is particularly suitable for producing 1 to 2 micron fiber at high rates and low cost. Each of the other thermoplastics possesses its own unique set of factors which influence operating costs.

The simplicity of this fiber-forming process is, in itself, of great importance. Not only can nonwoven fabrics and papers be made in a direct one-step operation, but complicated forms can be covered by simply directing the fiber stream toward the surface to be covered. For example, a screen mold can be used to make a glove shape, and then the fibers stripped off and the shape retained.

CONCLUSIONS AND RECOMMENDATIONS

A technique has been developed at NRL for the manufacture of submicron-diameter fibers from a variety of thermoplastic materials. The products, which may take the form of either loose fiber webs or tightly compressed sheets resembling cellulosic paper, may be used as specialty papers, nonwoven fabrics, and high-efficiency filter papers. When the fiber webs are bonded by impregnating resins, high strength is achieved, and when about 10 percent of an elastomeric binder is added, a highly porous synthetic leather results.

Since by selection of proper materials almost any combination of thermal and chemical resistance might be attained in the fiber webs, it is recommended that further study of binding agents be conducted. Particular emphasis might be placed upon the nonwoven and porous leather types. Combinations of fine organic fibers with coarser ones and with glass fibers hold interesting possibilities.

ACKNOWLEDGMENTS

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APPENDIX A

Design and Construction of Equipment

In the initial experimental design, a small nozzle consisting of 15 individual orifices that formed a row $3/8$ inch wide was supplied with compressed air heated inside $1/4$ -inch tubing located in the flue of a gas burner. Many design modifications were evaluated on this model. The nozzle was mounted on a 1-ounce Van Dorn injection molder, and the hydraulically operated ram-feeding mechanism was used to supply molten polymer.

Although the final equipment was developed from the original small-scale experimental design, it was scaled up to give 192 individual orifices in 4 groups, each 1 inch wide and separated by 1-inch spaces for clamping screws. This machine, which is supplied with electrically heated air, can produce a uniform fiber web in a continuous strip 9 inches wide. Figure A1 shows a diagram of the final apparatus; photographs of the extruder and its accessory equipment appear in Figures A2 and A3.

The most critical design feature of the apparatus is, of course, the nozzle for the molten polymers and the air (Figures A4 and A5); a detailed sketch is shown in Figure A6. To facilitate accuracy and uniformity in cutting the fine grooves, the extruder nozzle was constructed of free-machining stainless steel (type 416). The extruder nozzles were roughed to shape, surfaces A and C (Figure A5) were finished, but $1/16$ inch of stock was left on surface B (Figure A4). Surface C of both halves was ground to a No. 16 finish, and then these two surfaces were clamped together with surface A facing upward. The two A faces were next ground to obtain absolutely flat surfaces. While still clamped in this manner the orifice grooves were cut with a sharp $3/8$ -inch end mill. Positioned at an angle of 45° to surface A, the end mill produced 90° V-shaped grooves that matched perfectly when the two A surfaces were folded together. The result was a series of orifices having square cross-sections which extended the length of the nozzle. Surface A was lightly ground to remove burrs.

Surface B was then finished, and care was taken to preserve the knife edge and sharp points formed along its intersection with surface A. The final cuts on surface B were extremely light. In a final operation, the burrs at the ends of each groove were manually removed.

The air nozzle was fabricated from Class 5 tool steel, and surface D (Figure A5) was given a ground finish.

A $4 \times 4 \times 9$ inch melting block made of cold-rolled steel fits between the nozzles and the injection cylinder and serves both as a temperature regulator and a distributing head for the molten polymer. It has 10 holes drilled in from each end to receive $3/8$ -inch electric cartridge heaters. The block was made in two halves and matching fan-shaped slots were cut in each half to a depth of 0.013 inch. The slot was 7 inches wide at the front of the block and narrowed to $1-1/4$ inches at the backside in order to correspond to the $1-1/4$ -inch internal diameter of the injection cylinder. A conical cavity ($5/8$ inch deep and with a $1-1/4$ -inch base diameter) was bored into the back of the block to allow a smooth flow of polymer from the injection cylinder into the slot. The internal surfaces of the block were chromium-plated for corrosion protection.

To prevent polymer from melting inside the injection cylinder, a water jacket was installed at its forward end, and a Teflon gasket between the cylinder and block was added to provide some heat insulation between the two parts. The 1-1/4 inch internal surface of the cylinder was chromium-plated. A standard hydraulic system is used to operate the ram-feeding mechanism, and a pressure-regulating bypass valve controls the oil pressure which in turn controls the rate of feed. Recharging the injection cylinder by retracting the ram to a position behind the feed hopper is performed intermittently and may be as frequent as once a minute or as seldom as once every 30 minutes. The operation itself is so fast, however, that the flow of polymer is not stopped but only momentarily reduced. In practice, the operator initiates the recharging operation when it is seen that the hydraulic piston and ram have neared their position of maximum travel. Preferably, however, this action would be taken automatically by having the piston contact a forward limit switch which would then operate the hydraulic valve through a relay and solenoid-valve system. A similar limit switch could be used to start again the forward motion of the ram.

A rate-of-feed indicator (Figure A1) was devised to show, by the height of a water column, the rate of ram movement; it may be calibrated to read in volume units per minute. This device is based upon a duplicator piston and a cylinder which is filled from a water reservoir. As the ram moves forward, water is forced through a small glass-fiber-packed resistor, and the back pressure is indicated by the water level in a glass tube. The duplicating piston and cylinder do not duplicate the exact volume of polymer that is being fed to the nozzles, but the piston does duplicate the linear motion of the feed ram and consequently forces a proportionate amount of water through the resistor. This auxiliary cylinder has a 1-inch internal diameter as compared to the 1-1/4-inch diameter of the main cylinder.

Air temperature is controlled by an "on-off" type temperature indicator (range 0 to 1000°F) which actuates two 9-kw air preheaters that have alloy-sheathed elements. An input controller can be set to regulate the percent of time in a 30-second cycle during which power is supplied to the heaters. If this input setting is properly made, the air temperature may be held between 10°F limits. Air temperature for both the upper and lower halves of the nozzle is recorded on a 4-point strip recorder which is actuated by iron-constantan thermocouples. One of the points of this recorder is a controlled point which is connected to one of two thermocouples in the melting block. The block is heated by 20 cartridge-type heaters, each of which has a capacity of 100 watts. In order to give a smooth temperature control curve of less than 10° variation, the power to the heaters is regulated not only by the control relay but by a 2-kva variable autotransformer.

The air for this process, which is supplied at 100 psi, passes through a flow meter and is then reduced to the desired pressure. A capacity of 30 cfm is available for the attenuation, but if the full number of air nozzles described herein is used, a capacity of 60 cfm would be desirable. A maximum pressure of only 35 psi at the air nozzle is obtainable with the 30-cfm capacity.

The fiber-collecting screen, which measures 15 inches in width, is supplied with suitable masks to reduce the width of the suction area so that it will correspond to the total length of the row of extruder nozzles. The screen and two chromium-plated 6-inch calender rolls are driven by a variable-speed unit which permits production rates between 0 and 8 ft per min. A 2-hp blower is used to pull air through the screen. To determine the optimum distance between the nozzle and screen, the collection unit was built on small tracks (Figures A7 and A8).

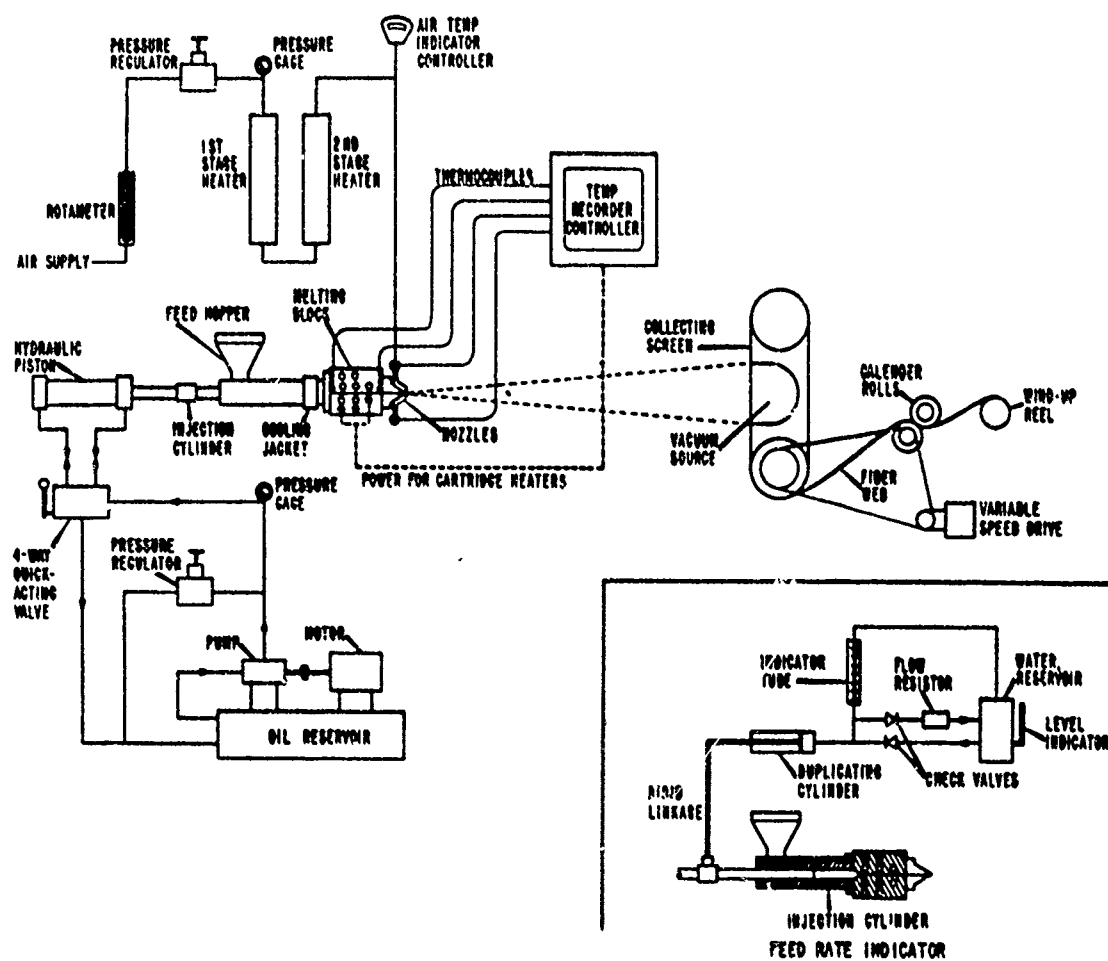


Figure A1 - Diagram of process equipment

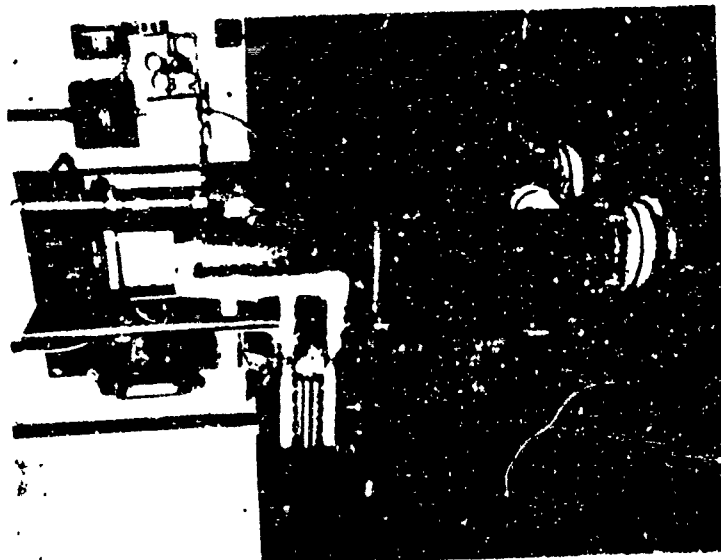


Figure A3 - Extruder and air heaters

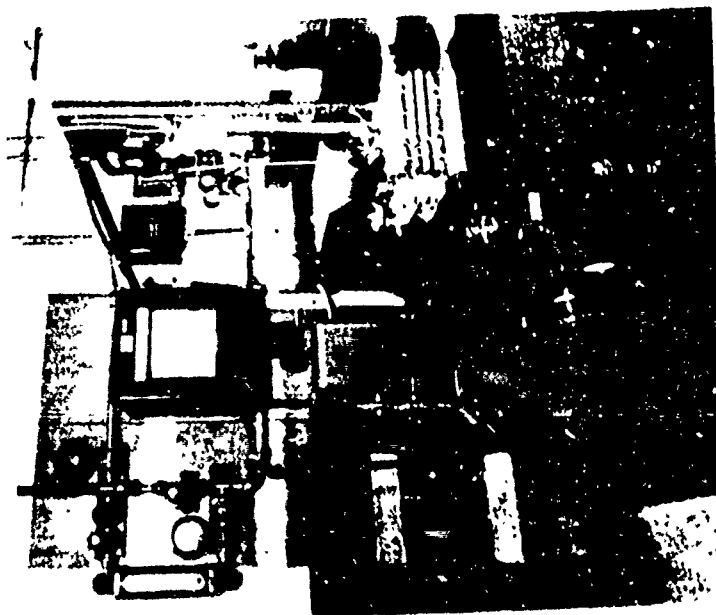


Figure A2 - Extruder and control equipment

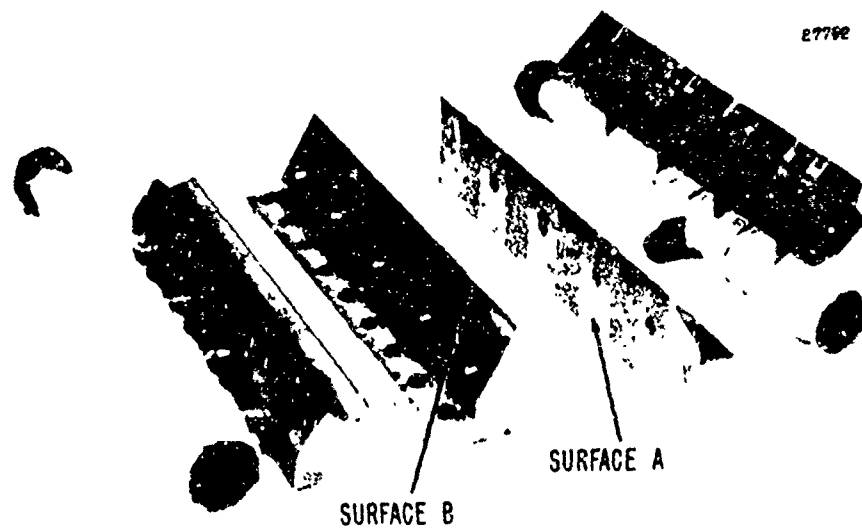


Figure A4 - Extruder and air nozzles (disassembled)

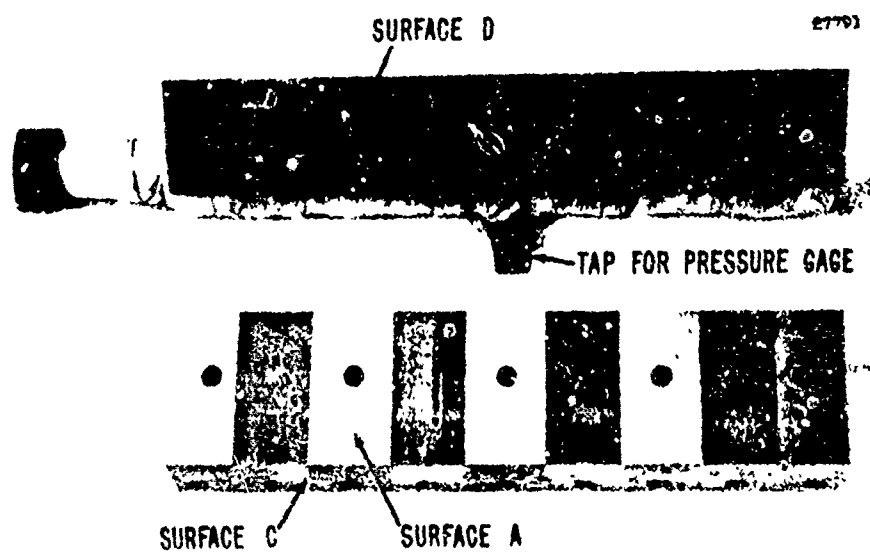


Figure A5 - Interior surfaces of nozzles

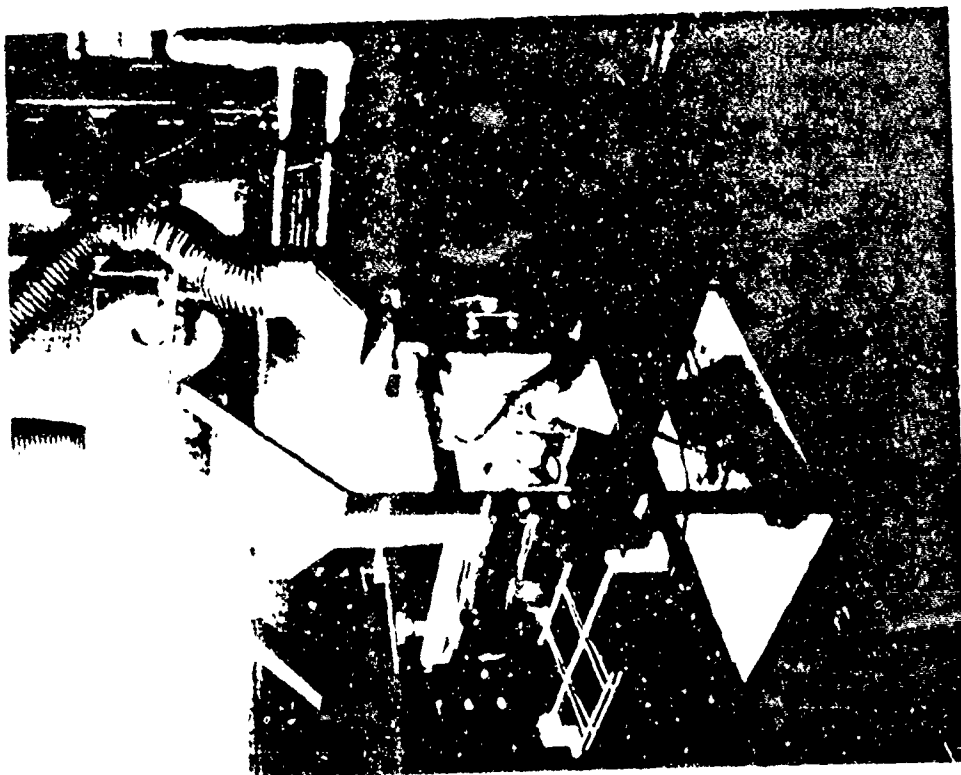


Figure A8 - Rear view of fiber-collecting screen



Figure A7 - Front view of
fiber-collecting screen

<p>UNCLASSIFIED</p> <p>Naval Research Laboratory. Report 4364. MANUFACTURE OF SUPERFINE ORGANIC FIBERS, by V. A. Wentz, E. L. Boone, and C. D. Fluharty, 15 pp. & figs., May 25, 1954.</p> <p>Through a new process recently developed at NRL, it is now feasible to produce submicron organic fibers from a variety of thermoplastic materials. By this method an adjustable extruder forces a hot thermo- plastic melt through a row of fine orifices into high- velocity dual streams of heated gas, usually air. The nozzle design provides for immediate resumption of attenuation following breaks, which are inevitable at submicron dimensions.</p> <p>Fiber diameters are determined by four basic process variables: air and nozzle temperatures, air (over)</p> <p>UNCLASSIFIED</p>	<p>I. Synthetic fibers - Manufacture</p> <p>I. Wentz, V. A. II. Boone, E. L. III. Fluharty, C. D.</p>
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